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Powder metallurgical components: Improvement of surface integrity by deep rolling and case hardening

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Abstract

In many cases, powder metallurgical components are heat treated in order to improve the component behavior. An established heat treatment technique for wrought materials is case-hardening. But up to now, this procedure is not applied for powder metallurgical components, since the existing porous microstructure avoids adjusting a defined carbon depth profile. As a consequence a carbonization of the core and a full hardening can occur, leading to a disadvantageous phase and residual stress distribution. However, a new approach for case hardening of powder metallurgical components is to perform a surface densification prior to the heat treatment. In this study this approach is realized by using a deep rolling process. In order to verify the functionality of the process chain, the surface integrity was analyzed after each processing step using X-ray diffraction measurements and materialographic methods. There is clear evidence that the open porous structure could be transferred into a surface densified graded porous layer. As a consequence the carburization is dominated by solid state diffusion enabling to adjust a defined carbon depth profile leading to a martensitic surface layer and compressive residual stresses in the near surface zone. Subsequent fatigue strength experiments for sintered samples as well as for additional densified or densified plus case-hardened specimens were performed in order to demonstrate the enhanced mechanical properties. A significant increase in fatigue strength can be measured.

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Keywords: powder-metallurgical components; heat treatment; rolling

1. Introduction

The powder metallurgical (PM-) route is a near net shape manufacturing process avoiding subsequent machining [1]. In the most basic case, the PM route is consisting of the steps compaction and sintering (see Fig. 1.).

This optimal utilization of material and high efficiency of the PM-process is opposed with the disadvantage of a porous microstructure. The existing pores affect the material properties and therefore the component behavior [1, 2]. Especially the mechanical properties are inferior to machined wrought components. Therefore the PM route is rarely not used for highly loaded components [2].

Fig. 1. Powder metallurgical route [1].

Recently, efforts are undertaken to use the PM-route for highly stressed components. Thereby three strategies can be identified. The first one is to add alloy elements to the base powder in order to increase solid solution effects. The second strategy is to use mechanical surface treatment processes to densify the surface layer and to induce strain hardening as well as compressive residual stresses. The third option is to perform a heat treatment process to change the ferritic-pearlitic structure and to

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induce strain hardening as well as compressive residual stresses.

Up to now, two-thirds of all PM-components are heat treated [3]. Case-hardening however, a standard technique for wrought components is not an established heat treatment technique [4]. This is due to the porous microstructure preventing the adjustment of a defined carbon depth profile. Both the surface carbon concentration and the case depth are increasing with rising porosity [4, 5]. Given a sufficient porosity there is the risk to carburize the core material [4]. For the further consideration a separation between an open interconnected porosity and an individual porosity beyond the surface is appropriate. In case of an open porous structure the carburizing is dominated by gas diffusion processes through the open porosity [3 - 5]. As a consequence, the mass transfer is shifted from the macroscopic and geometric surface to the surface area of the pore channels. One might even call it an internal carburizing process. Thus, solid state diffusion processes become secondary and there is the risk to carburize the core of the PM-component [5 - 8]. But even if there is a closed porous structure, an accelerated carbon diffusion can be detected, although the mass transfer occurs along the macroscopic surface and gas diffusion processes can be excluded [4]. The reason for this is that surface diffusion processes can occur in addition to volume diffusion [9 - 10]. The diffusion coefficient for atomic transport along the free pore surface is several magnitudes larger than through the bulk material. In the case that the free pore surface is orientated in diffusion direction, the carbon penetration depth is increasing. [11].

One approach for case hardening avoiding a carburizing of the core is to transfer the open porous structure into a surface densified layer prior to the case-hardening process. One option for a surface densification process is deep rolling. A possible process chain is then given in Fig. 2.

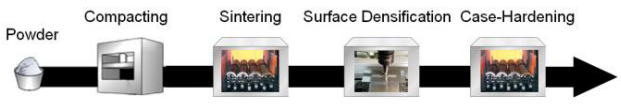

Fig. 2. New approach for case hardening of powder metallurgical components is surface densification prior to heat treatment.

In this publication, the surface integrity after every process step is documented. The investigations are focused on the porosity, carbon, phase and residual stress distribution. In order to demonstrate the enhanced mechanical properties, alternating bending tests were performed to determine the fatigue strength after every process step.

2. Material and Sample Geometry

The base material for this study was Astaloy 85 Mo, a pre-alloyed, water atomized powder of Höganäs AB. The chemical composition of this commercial powder is given in Table 1.

Table 1. Chemical composition of Astaloy 85 Mo.

element	C	Mo	Mn	Si	Cu	Cr
[wt%]	0.01	0.866	0.128	0.009	0.0026	0.038

For this investigation, the almost carbon free Astaloy 85 Mo was admixed with graphite in order to increase the carbon concentration to 0.25 wt%.

The specimen for the investigation was a fatigue strength (FS-) bar. The geometry is given in Fig. 3.

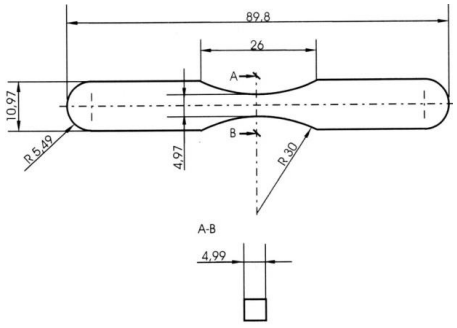

Fig. 3. Technical drawing.

This specimen was manufactured by the powder metallurgical route. Sintering was performed for 20 minutes in a nitrogen based atmosphere at 1120 °C resulting in a density of 7.2 g/cm³. In the following, such components are named as-sintered FS-bars.

Two-thirds of all sintered samples were additionally deep rolled by a deep rolling tool type HG 6 of Ecoroll AG (Germany). The used parameter set is given in Table 2. The deep rolling was limited to the waisted zone of the FS-bar and was carried out in a meandric way. Hereinafter these specimens are named surface densified FS-bars.

Table 2. Chemical composition of Astaloy 85 Mo.

parameter	Value
Distance between meandric paths [mm]	0.04
Feed rate [mm/min]	1000
Rolling pressure [bar]	180

Half of all surface densified FS-bars were case-hardened using a bell furnace of type SOLO 202 at the IWT Bremen, Germany. The heat treatment was performed at 940 °C using a maximum carbon potential

of 1 wt%. The carburizing procedure takes 2 hours and the carbon potential during the carburization is shown in Fig 4. For the subsequent quenching step an oil bath was used. In order to avoid a carburization of undensified material domains, these zones were masked by the stop-off paint Vaccucoat 0095 (Nüssle GmbH, Germany).

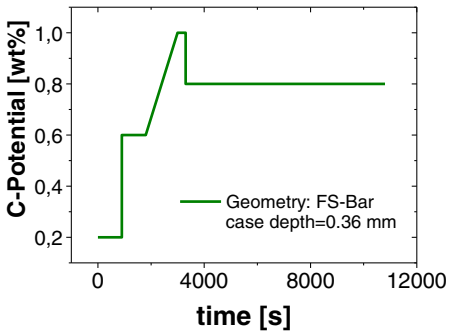

Fig. 4. Carbon potential in the furnace during carburizing.

Ultimately there are three different type of specimens, namely the as-sintered, the surface densified and the case-hardened FS-bar.

3. Testing and Analyzing Procedure

3.1. Porosity Distribution

The porosity distribution was measured by image analysis using the commercial software package analySIS pro (Olympus Stream, Japan). For characterization, the porosity was opened by a grinding and polishing treatment according to recipe given in [11, 12]. Subsequently the pores were infiltrated and materialographically prepared once more [11]. The threshold value for the image analysis was automatically determined by the software analySIS pro.

3.2. Carbon Distribution

The local carbon concentration was detected by optical emission spectrometry (OES) with a spark spectrometer of type ARL 3640 at the IWT in Bremen (Germany). A depth profile could be determined by alternating grinding and measuring processes.

3.3. Microstructure Distribution

The sample preparation is based on the idea to open the porous structure and infiltrate the pores. After this preparation a final grinding and polishing step was performed. A detailed instruction is given in [11, 12]. The polished sections were etched by a double etching technology using Nital and Pikral.

3.4. Residual Stress Distribution

The residual stress measurements are performed by X-ray diffraction using CrK_{α}-radiation and a diffractometer in ψ-configuration. The residual stresses were evaluated using the $\sin^2\psi$-method. The elastic constants were $E^{\{211\}} = 219911$ MPa and $\nu^{\{211\}} = 0.28$.

The determination of a residual stress depth profile is possible by alternate measuring and electrochemical polishing procedures. Residual stress redistributions during electrochemical polishing are not considered. Each state was measured once.

3.5. Fatigue Strength

Alternating bending tests were performed to determine the fatigue strength using a bending testing equipment of design Schenck (Germany). The tests were performed according to the description in [12]. A 20 % drop of bending stresses or 10^7 load cycles without a failure serve as termination criterions. The evaluation of the experimental data was done by the arcsin√P-method. For each S-N-curve at least 25 specimens were used.

4. Results

4.1. Porosity Distribution

The parameters of the PM-route in this study result in an initial density of 7.2 g/cm³ for the as-sintered FS-bars. This is equivalent to an average porosity level of ~ 7.5 %. Due to the mechanical surface densification by deep rolling the near surface layer is densified (see Fig. 5).

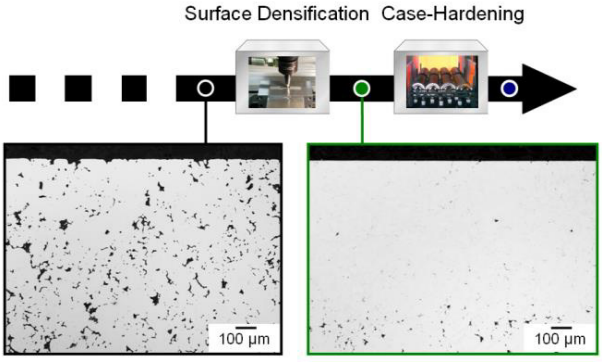

Fig. 5. Microstructure before and after the deep rolling process.

The first 250 µm are almost pore free (see Fig. 6). The total depth effect of the mechanical surface densification process can be specified with 750 µm until the initial porosity level can be measured. The subsequent case-hardening process does not influence the adjusted porosity depth profile. Thus, the porosity depth profile of the surface densified FS-bar is also valid for the case-hardened specimens.

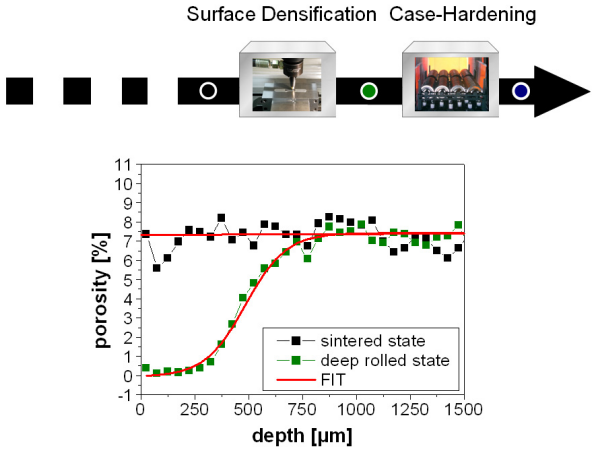

Fig. 6. Porosity depth profile of powder metallurgical FS-bars after subsequent deep rolling.

4.2. Carbon Distribution

Initially the commercial Astaloy 85 Mo powder was admixed with graphite. Thus, the as-sintered as well as the additionally surface densified specimens have a uniform carbon concentration of 0.25 wt%. Due to the subsequent thermo-chemical carburizing step the surface layer is enriched with carbon (see Fig. 7).

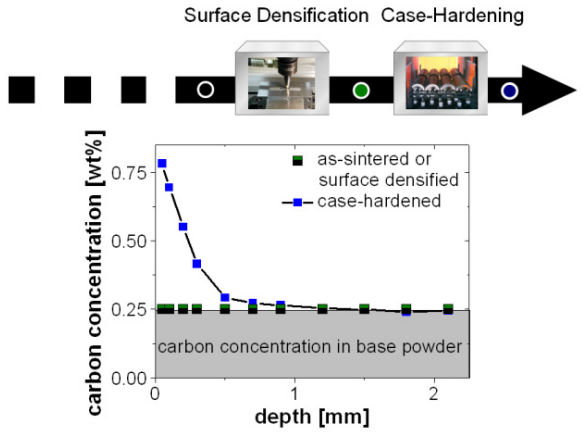

Fig. 7. Carbon depth profiles before and after the case-hardening step.

A maximum carbon concentration of 0.78 wt% at the surface was measured. The total carbon penetration depth was 750 µm. At greater depths the initial carbon concentration of 0.25 wt% was detected.

In conclusion, a defined carbon depth profile could be adjusted by having a surface densification prior to the case hardening process. As a consequence the mass transfer is shifted to the macroscopic surface and the carburizing is dominated by solid state diffusion processes.

4.3. Phase Distribution

In the as-sintered FS-bars, a ferritic-pearlitic dominated microstructure exists, since the cooling rate after sintering was low and the base material is characterized by a homogenous alloying element distribution.

As a consequence of the thermo-chemical process, a martensitic surface layer is formed, see Fig. 8. The thickness of this layer can be specified with 0.5 mm. The following layers as well as the core material have a bainitic structure. Since the used material is dominated by molybdenum, no ferrite or pearlite fractions could be found.

The left and the right side of the cross-section of Fig. 8 were masked with the stop-off paint Vaccucoat 0095. As a consequence, no carbon mass transfer could occur and therefore no martensite was formed at these sites.

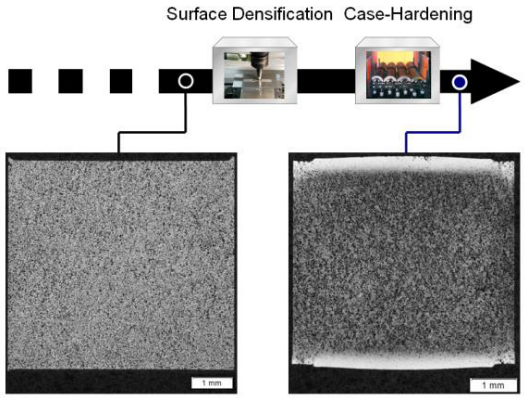

Fig. 8. Phase distribution in a cross section after sintering and after the thermo-chemical treatment.

4.4. Residual Stress Distribution

The residual stress distributions are shown in Fig. 9. The profile of the as-sintered specimen indicates that there are hardly any residual stresses detectable. The as-sintered FS-bars are therefore regarded as free of

residual stresses. In contrast, both the densified and the case-hardened FS-bars have compressive residual stresses in the near surface layer.

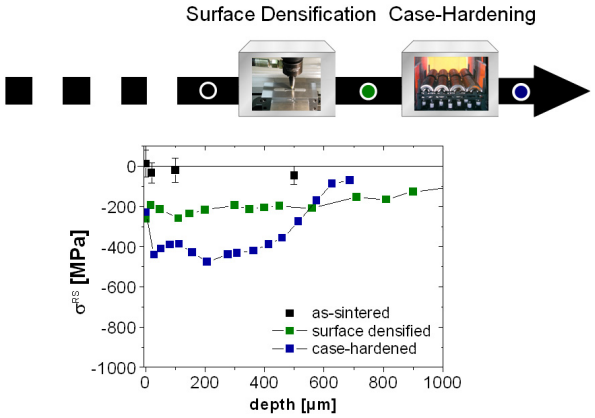

Fig. 9. Residual stress distribution of the as-sintered, the surface densified and the case-hardened FS-bar. Redistribution effects are not taken into account. Each state was measured once. Maximum uncertainty of measurement: 60 MPa.

Fig. 9 shows that the compressive residual stresses of the heat treated FS-bars are even more significant than in the just surface densified component state. The maximum is -446 MPa in a depth of 110 µm for the case-hardened compared to -259 MPa in a depth of 200 µm for the surface densified FS-bar. However, comparable residual stress values of -229 MPa for the case-hardened and -261 MPa for the surface densified FS-bars could be measured at the surface. This means, that the compressive residual stresses of the case-hardened specimens are strongly reduced at the surface. Reason for this is an internal oxidation due to the gas carburizing process. The results indicate that the zero-crossing of the residual stresses of the additionally case hardened samples are in smaller depth compared to the profile of the surface densified specimens. In all cases, redistribution effects are not taken into account and therefore no zero crossing could be detected. As a consequence the surface densified FS-bars seem to have a compressive residual stress state up to a depth of 1000 µm.

4.5. Fatigue Strength

Alternating bending tests were carried out to determine the fatigue strength using at least 25 specimens per material state. For the as-sintered FS-bars a fatigue strength (fracture probability of 50%) of 125 MPa was determined. The additionally performed deep rolling process and the associated densification of the surface layer will increase the capacity to withstand bending stresses to 250 MPa, see Fig. 10. A subsequent thermo-chemical treatment induces higher compressive residual stresses as well as a martensitic surface layer and further enhances the mechanical properties. FS-bars manufactured according to this process chain have a fatigue strength of 475 MPa. In summary, the fatigue strength can be improved by a factor of 3.8 using the process chain shown in Fig. 2.

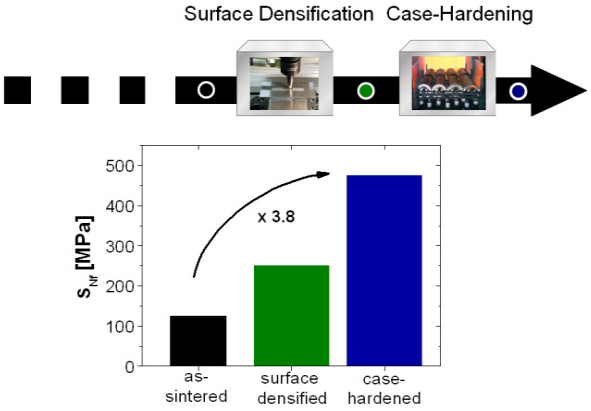

Fig. 10. Results of alternating bending tests. Fatigue strength of as-sintered, surface densified and case-hardened FS-bars.

5. Discussion

The as-sintered specimens are characterized by a homogenous distribution of porosity and alloying elements. Since the density is lower than 7.5 g/cm³, an interconnected porosity can be assumed. Due to the low carbon concentration and a low cooling rate after the sintering process, a formation of martensite and bainite can be excluded. As a consequence, the microstructure is ferritic-pearlitic and the as-sintered component state is almost free of residual stresses. Thus, the as-sintered FS-bars have very poor mechanical properties.

The open porous structure including the interconnected porosity in the surface layer can be transferred into a densified zone by carrying out a deep rolling process. Simultaneously, compressive residual stresses due to inhomogeneous plastic deformation as well as work hardening are induced in the surface layer. Therefore, the load capacity of the surface layer is increasing. In this study, the fatigue strength is increased by a factor of two. In [13] the effects of densification, residual stresses and work hardening were separated and quantified. It was shown that the main effect was caused by work hardening. Surface densification and residual stresses played a subordinate role [13].

In case of the case-hardened specimens, the surface densified FS-bars were additionally thermo-chemical treated. Since there is no open and interconnected porosity, a carburizing step can be performed without the risk to carburize the core of the FS-bars. The mass transfer is then limited to the macroscopic surface and the dominating atomic mass transport mechanism is soild state diffusion. The results of chapter 4 confirm this by showing a defined carbon depth profile. Since a defined carbon distribution can be adjusted, a chemical stabilization of the austenite occurs and diffusion controlled phase transformations are no longer possible due to too low driving forces during the rapid cooling. As a consequence, the highly alloyed surface layer forms a carbon supersaturated martensite by a diffusionless transformation based on shear deformations. Thus, an increase in the dislocation density occurs. Since a lower carbon concentration of the core is leading to an earlier phase transformation based on diffusion processes with a simultaneously lower increase in volume compared to the martensitic reaction, compressive residual stresses are induced in the surface layer. Alltogether, the densified surface zone, the increase in work hardening, the compressive residual stresses and the martensitic structure are leading to optimal mechanical properties. The fatigue strength is 3.8 times higher compared to the initial as-sintered specimens.

6. Conclusion

Due to a surface densification process prior to the thermo-chemical treatment, a defined carbon depth profile in powder-metallurgical components can be adjusted. This enables to form martensite and compressive residual stresses in the surface layer. The reduced porosity in the surface layer, the martensite as well as the compressive residual stresses enhance the mechanical properties, especially in case of a highly loaded surface. The fatigue strength can be increased significantly by the presented process chain. Thus, the PM-route seems to be applicable for highly stressed components.

Acknowledgements

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